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MEMBRANE VAPOR ENRICHER FOR WATER FOR INJECTION (WFI) PRODUCTION

Phase 1 Final Report

Dr. Arye Gollan
Myles H. Kleper

December 15, 1987

Supported by

U. S. ARMY MEDICAL RESEARCH AND DEVELOPMENT COMMAND
Fort Detrick, Frederick, Maryland 21701-5012

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A/G Technology Corporation
34 Wexford Street
Needham, Massachusetts 02194-2912

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The US Armed Forces have a logistical requirement for a continuous demand, high purity, Water For Injection (WFI) system to alleviate the logistical burden of transport and maintenance of large supplies of parenteral solutions. In this program, the feasibility of incorporating permeable membrane gas separation technology as the key element in the WFI production process has been evaluated. These high productivity, imperfection-free gas permeable membranes are highly selective to water vapor and are packaged in compact and lightweight hollow fiber module elements.

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FOREWORD

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I. EXECUTIVE SUMMARY

To alleviate the existing necessity to carry and maintain large supplies of parenteral solution in bulky, dated plastic bags, a joint effort by the Army, Navy and Marines, and Air Force has been undertaken to develop a Resuscitation Fluids Production System (REFLUPS) to produce Water for Injection (WFI). The WFI system must be easily transportable, self-contained, energy-efficient, require minimal operator attention and be exceptionally reliable. The REFLUPS should produce a nominal volume of 75 liters/hour (+/- 20%) of WFI.

A new concept for WFI production has been evaluated in this Phase 1 feasibility program. This WFI system is based on the advanced permeable membrane technology developed by A/G Technology Corporation for gas separation. These membranes have exceptional productivity for water vapor, high water vapor to air selectivity and are available in compact yet rugged hollow fiber cartridge elements.

Two approaches were evaluated for WFI production:

1. Water Vapor Enrichment from a Gas Stream Produced by Aeration/Evaporation of the Source Water; and,

2. Water Vapor Enrichment from a Heated Source Water.

The enriched water vapor stream, in both cases, was then condensed to produce WFI.

The first approach (and the original concept for WFI production) was shown to produce exceptionally high quality water from a variety of source waters ranging from tap water to brackish water to waste water. Even though the test system was not constructed to medical grade standards, product water quality was high with a pH of 6.5, turbidity of 0.1 to 0.5 NTU, conductivity of 2 to 5 μmhos/cm and a TOC of 2 to 3 mg/liter. Typical productivity at 160 F for this operating mode was 0.08 liters/ft^2 of membrane area/hour.

Technical direction from the Project Monitor suggested that the source water for WFI production would be of tap water quality, hence the possibility of processing hot water through the compact hollow fiber membrane module in a "pervaporation" mode became viable. This mode of operation should produce WFI of similar quality to the vapor processing mode. Furthermore, it was demonstrated to exhibit about a 3-fold higher productivity than the first approach, producing 0.23 liters/ft^2 of membrane area/hour (160 F).

The pervaporation mode also simplifies system design A first step aeration/evaporation column is not required and, since the volume of non-condensibles permeating the membrane is reduced, the down stream vacuum pump capacity is lowered.

The productivity of the permeable membrane vapor enricher was stable during an extended duration test which covered 80 hours over a test day operating period.

Optimization of the pervaporation approach has not yet been performed, nor have the lightest weight process components been identified. Nonetheless, a preliminary conceptual design indicates that a WFI system , excluding instrumentation, based on the A/G Technology gas permeable membrane elements will weigh between 180 and 340 lbs (dependent on condenser type, air or water cooled), require 20 to 30 ft^3 volume and utilize 0.8 to 1.25 hp with available waste heat. System designs for self-contained units incorporating a heat pump to eliminate the need for waste heat will add minimal weight and volume but are projected to increase the power requirement by about 5 hp.

System optimization and bread-board system testing are planned in the Second Phase of this project.

II. INTRODUCTION

A. Statement of the Problem

The US Armed Forces have a logistical requirement for a continuous demand, high purity, Water For Injection (WFI) system capable of operation on the range of surface or drinking waters encountered in remote locations and/or combat situations. The WFI system must be easily transportable, self-contained, energy-efficient, require minimal operator attention and be exceptionally reliable. To date, no suitable WFI system has been put into service although a multi-stage, serial membrane filtration system is under development.

To alleviate the existing necessity to carry and maintain large supplies of parenteral solution in bulky, dated plastic bags, a joint effort by the Army, Navy and Marines, and Air Force has been undertaken to develop this Resuscitation Fluids Production System (REFLUPS). The REFLUPS should produce a nominal volume of 75 liters/hour (+/- 20%) of WFI to the following standards:

(a) US Pharmacopeia (USP) XXI,
(b) Water for Injection (WFI) Standards, page 850, and
(c) UL 544, Standard for Safety, Medical and Dental Equipment.

B. Phase 1 Program Approach

A new and novel concept for WFI production has been evaluated in the Phase 1 feasibility program. This WFI system is based on the advanced permeable membrane technology developed by A/G Technology Corporation for <u>gas separation</u>. As explained in detail subsequently, the proposed system consists of three principle, straightforward steps:

1. <u>Aeration/Evaporation</u> (utilizing waste heat) to Produce Water Vapor from any Source Water.
2. <u>Gas Separation Membrane Processing</u> to Produce a Water Vapor Enriched Stream, Free of Contamination.
3. <u>Condensation/Deaeration</u> to Produce WFI and Remove Non-Condensibles.

Final product water polishing (e.g., activated carbon adsorption) is not anticipated to be required for organics removal, based on the Phase 1 testing with simulated waste waters (i. e., worst case conditions). If necessary, such polishing is straightforward and can be added at a latter date. As such, carbon adsorption post-treatment was not a subject of concern in the feasibility portion of this program.

The above approach was predicated on the belief that the WFI system, under extreme circumstances, might be utilized with contaminated source waters or waste waters in combat situations. It was learned subsequent to the initiation of Phase 1 Testing that the incoming water to the WFI system will be of essentially tap water quality (1). Operation with 'clean' water <u>eliminates</u> the need for first step in the process: aeration and evaporation, since high organics loadings do not need to be stripped out of the water and since membrane surface fouling or fiber plugging are no longer of concern. Thus, rather than passing water vapor through the membrane cartridge for gas/gas separation (water vapor/air), hot water can be processed in a pervaporation process for liquid/gas separation (water/water vapor). The pervaporation approach not only eliminates the need for the aeration/evaporation contact column and air blower but it also reduces the load on the system condensation/deaeration step since there is a greatly reduced non-condensible loading.

The pervaporation approach is thus summarized as:

1. Preheating (utilizing waste heat) of the Source Water.
2. Pervaporation Across a Gas Separation Membrane to Produce a Water Vapor Enriched Stream, Free of Contamination.
3. Condensation/Deaeration to Produce WFI and Remove Non-Condensibles.

The preheating step and the condensation step may be able to be integrated with the existing environmental control system. Alternatively, a self-standing unit incorporating a heat pump for circulating a refrigerant to heat the incoming water and cool the water vapor enriched stream is practical.

The key to system success with either approach is the gas separation membrane. This membrane is defect-free to provide bacteria, virus and pyrogen-free water; has a high transport for water vapor to reduce membrane area requirements; is provided in the compact hollow fiber membrane cartridge form; is capable of operation at elevated temperatures sufficient to suppress bacterial growth (perhaps up to autoclaving conditions); and, is storable in a dry state with no break-in period.

The ability to operate at high temperatures is especially important in the operation of equipment producing WFI since system contamination by bacteria would be highly detrimental.

The advanced AVIR™ permeable membranes for gas separation developed by A/G Technology have been shown to have a superior combination of gas selectivity and gas permeability versus other commercial and developmental membrane products for oxygen/nitrogen separation (2,3). Preliminary test data at ambient temperatures, which served as a basis for program initiation, suggested that these membranes have at least a 10-fold, and perhaps a 100-fold, higher water vapor/air separation factor and water vapor transport rate across the membrane than published data for other available gas separation membrane elements (3, 5).

In the Phase 1 feasibility program, the potential of both of these novel approaches to WFI were assessed. REFLUPS development, design and prototype testing will be incorporated into the Phase 2 and Phase 3 programs.

III. MEMBRANE SEPARATION PROCESSES BACKGROUND

A. General

Membrane separation technology has gained wide acceptance throughout industry and the Armed Services for a range of liquid/solid and gas/gas separations. The relationship between the major membrane technologies of Microfiltration, Ultrafiltration, Reverse Osmosis and Gas Separations is depicted in Figure 1.

Microfiltration is customarily referred to as the separation or removal of particles larger than 0.02 micron and less than 10 micron. The separation provided by microfilters is inherently physical in nature. Classical microfilters are often referred to as "absolute" filters for a given particle size, meaning they demonstrate 100% removal ability for such particles starting from time zero.

Ultrafiltration membranes separate suspended, colloidal and dissolved species on a molecular basis. A number of nominal ultrafiltration membrane pore sizes are available throughout the range of about 2,000 daltons to 500,000 daltons. As with microfiltration membranes the separation is based on pore size, however the build-up of a solids layer at the membrane surface (concentration polarization) may serve to increase membrane retention and decrease membrane productivity. The vast majority of ultrafilters offered commercially are not "absolute" since these membranes are characterized by large pores ("pinholes") produced in the manufacturing process.

Small dissolved species such as sugars and salts (nominal molecular weight down to about 100 daltons) can be removed from aqueous streams by reverse osmosis. Separation by reverse osmosis is primarily through a "solution-diffusion" mechanism, in which the permeability of the membrane to any constituent is proportional to the product of its solubility in the membrane and its diffusivity through the membrane. The nature of reverse osmosis membranes is such that they are not absolute filters and bacteria passage can occur. This type of membrane is also subject to a high degree of fouling due to scaling by sparingly soluble salts and a range of potential "bad actors" in the feed stream. Hence, pretreatment to reverse osmosis is always extensive. Finally, it is of note that organics rejection by reverse osmosis is typically low, potentially due to the high binding affinity of organics to water.

B. Gas Separation Background

Gas separations in polymeric membranes, in the absence of any bulk reaction, occur due to different levels of solubility in, and diffusion through, the separating layer (4). Solubility being a thermodynamic property while diffusion is a kinetic quantity. The permeability, P_r, of a given gas is an intrinsic property of the membrane. It is closely associated with Henry's Law for simple and non-interacting gases, and for low concentration levels is given by

$$P_{ri} = S_i \times D_i \qquad [1]$$

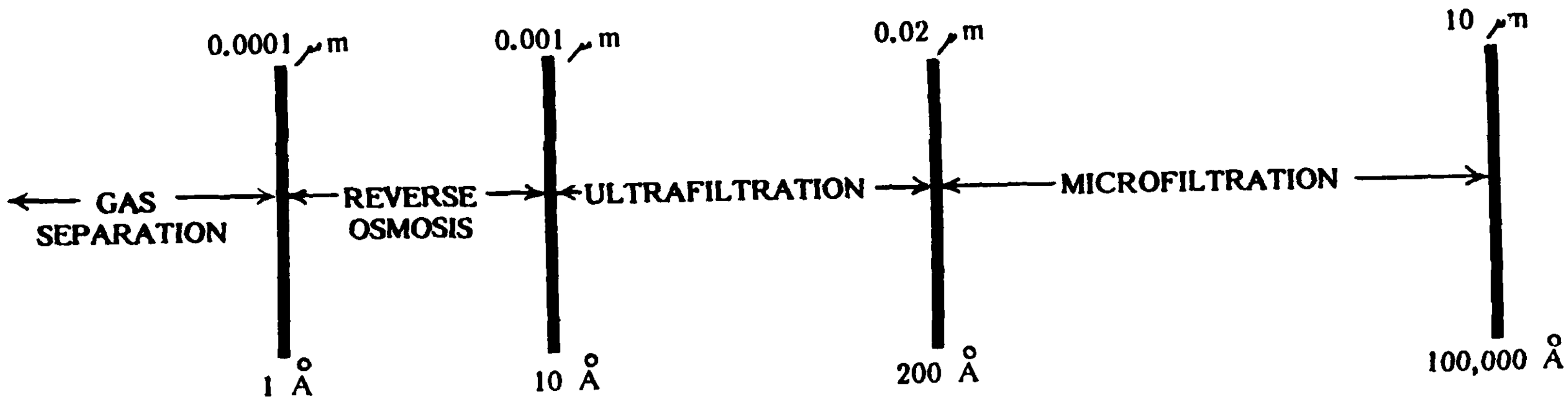

Figure 1.

Range of Separation for Membrane Processes where,

P_r = Intrinsic Permeability of gas i in the membrane, cm^3 (STP) cm/cm^2 sec cm-Hg.
S_i = Solubility of gas i in the membrane, cm^3(STP)/cm^3 cm-Hg.
D_i = Diffusivity of gas i in the membrane, cm^2/sec.

The mass transport of gas through a membrane is given by Fick's Law:

$$N_i = \frac{P_{ri}}{d} A \ DP \qquad [2]$$

where,

N_i = Flowrate of gas i, cm^3(STP)/sec
A = Membrane area, cm^2
d = Membrane separating barrier thickness, cm
DP = Transmembrane partial pressure difference of gas i, cm-Hg

For a given membrane system, the degree of separation between gases will depend upon the relative permeabilities of the gases to be separated. This ratio of gas permeabilities is termed the <u>separation factor</u>, or membrane <u>selectivity</u>, and the larger the value of this factor, the better the separation. For the separation to be economically feasible the flowrate, N, through the membrane must be sufficiently high.

By convention, the enriched gas stream passing through the membrane is termed the **permeant**, while the gas stream remaining on the feed-side of the membrane is termed the **retentate.**

It follows from Equation [2] that for a given membrane area the intrinsic permeability, P_{ri}, is not the only parameter contributing to the gas flowrate through the membrane. Equally important is the membrane thickness and the driving force across the membrane (DP). For economical and energy conservation reasons, the driving force must be kept as low as possible.

Thus, polymers which have high intrinsic permeability values and good separation factors are not necessarily suitable for commercial membrane systems if they cannot be rendered into sufficiently thin layers. What must be taken into account is the <u>membrane effective permeability</u>, P_{ri}', defined as:

$$P_{ri'} = \frac{P_{ri}}{d} \qquad \frac{cm^3 \text{ (STP)}}{cm^2 \text{ sec cm-Hg}} \qquad [3]$$

Dense membranes have a uniform membrane separating layer throughout their thickness and, therefore exhibit inherently low productivity. Asymmetric membranes, in which a thin "skin" layer is supported by an integral backing and composite membranes which have a thin coating added to the support to perform the separation, have been developed to provide higher effective permeability values.

To perform gas separations most economically, the membrane selectivity must be high enough to achieve the degree of separation required under realistic operating conditions while the membrane effective permeability must be sufficiently high to reduce the membrane area requirement which is directly related to the system capital cost.

C. Pervaporation

Pervaporation involves diffusional flow through a polymeric barrier (Perm-selectivity) and evaporation/distillation separation. It is, therefore, a two-step separation wherein a liquid is flowed on one side of the membrane and vapor is permeated across the membrane. Pervaporation has demonstrated exceptionally high separation factors between two components and is generally very effective in separating azeotropic mixtures.

As in all systems that exhibit very high selectivity, imperfections in the separating barrier are absolutely unacceptable due to liquid carryover interference with the separation process.

D. Differences Between Liquid and Gaseous Membrane Systems

There are a few major differences between membrane separations based on liquid/solid separations versus gas/gas or gas/liquid separations. When processing gases or performing pervaporation separations:

- -- Concentration polarization becomes less significant,
- -- Operating pressure sensitivity increases (gas/gas separations),
- -- Feed concentration sensitivity decreases, and
- -- The impact of membrane imperfections becomes more pronounced.

In gas processing the rate of mass flow/unit area is considerably lower than for liquids and the diffusion of molecules from the membrane surface to the bulk stream is significantly higher. Therefore, concentration polarization, which is a key concern in reverse osmosis is not as big a factor in gas treatment. In designing for gas treatment, gas flow rates should be chosen to minimize module pressure drop and to provide good distribution in the membrane module.

Gas/gas membrane separations are considerably more sensitive to operating pressures than liquid systems but are generally less sensitive to feed concentration. Since gas systems do not have to overcome feed osmotic pressure to operate efficiently, low pressure systems (< 75 psig) or vacuum systems can be designed providing a high effective permeability gas membrane is available.

The impact of imperfections on the membrane separation is more pronounced in gas processing and pervaporation due to the large ratio of hydraulic mass flow through an imperfection to the flow of gas through the membrane. Gas separation membranes must be produced defect-free or the ability to repair (or plug) defects with a coating material is required. The severity of the defect problem increases, the higher the selectivity of the membrane. Since gas separation membranes are based on molecular diffusive transport, gas separation membranes must have the tightest molecular aggregation state of any membrane technology. It follows therefore, that imperfections in gas membranes are unacceptable and, unlike reverse osmosis membranes, they are inherently "absolute" filters for bacteria, virus and pyrogens.

E. Gas Membrane Cartridge Geometry Options

Since the permeate gas flow per unit membrane area is inherently low, a compact membrane cartridge design is required if economical system designs are to be realized. The two membrane cartridge options with the best packing densities are the spiral wound and hollow fiber geometries.

Spiral wound cartridges consist of a number of "leaves" each containing two flat sheets of membrane separated by porous support material. Spiral wound cartridges are reasonably compact and system designs incorporating spirals require simple pressure vessels. Gas by-pass around the cartridges due to "brine seal" misalignment or failure within the pressure vessel is a potential concern which may reduce spiral wound cartridge productivity. Also, spiral wound cartridges are usually operated in a cross flow mode which tends to slightly lower the achievable gas separation levels for a given set of membrane transport characteristics. Additionally, the spiral wound cartridge has a significant length of glue seams which pose a greater potential for permeate contamination by feed gas than is present in some competitive module geometries.

Hollow fiber technology provides the cartridge of choice for membrane gas separations. Hollow fiber cartridges have the best packing density, by far, and are simple to operate and maintain on clean gas streams. The fact that membrane fibers are self-supported and can operate at up to several hundred psi pressure differential greatly simplifies cartridge design and assembly options.

Hollow fiber cartridges have significantly better packing densities than spiral wound cartridges. A typical 4-inch diameter spiral wound cartridge designed for liquid treatment contains about 20 ft^2 of active membrane area per foot length. Hollow fiber cartridges with identical outside diameters will have, about 100 ft^2 of active membrane area per foot length in practice (0.6 mm OD fibers, 50% void space). In addition to this 5-fold increase in membrane area, the hollow fiber cartridge is provided in its own pressure vessel and does not require a larger pressure vessel for operation as is necessary with spiral wound modules.

Hollow fibers will also be the configuration of choice for a WFI system based on pervaporation, since compactness, minimal weight and good flow distribution are critical.

F. Candidate Membranes for Water Vapor Enrichment

The proposed concept for WFI production is based on the ability of gas separation membranes to enrich the water vapor content in an air stream. The degree of enrichment is important to the extent that the permeant stream should have minimal non-condensibles (oxygen and nitrogen) since these will have to be pumped out of the system at an increased energy penalty. Equally important is the membrane effective permeability (or transport) for water vapor, since high water vapor transport minimizes system weight and volume.

A number of different membrane materials are potentially suitable for water vapor enrichment from air, although limited data are available. A summary listing of the membrane materials, membrane geometry, and available data for either oxygen, nitrogen or water vapor transport is provided in Table 1. Although, the data are limited, one can extrapolate the water vapor transport from the oxygen/nitrogen data since, most commonly, water vapor transport is two to three orders-of-magnitude higher than oxygen/nitrogen transport.

From Table 1 it is clear that only two membrane types can offer the potential high degree of water vapor transport necessary to provide an efficient, compact WFI system. These membranes are the advanced permeable membranes of A/G Technology and the silicone-based membranes offered by UOP (and others). Silicone-based membranes, however, have about one-tenth the selectivity estimated for A/G Technology membranes. Therefore large volumes of oxygen and nitrogen (non-condensibles) would be permeated along with the water vapor increasing the energy demand of the system. More critically, silicone-based membranes, being rubbery polymers, have less tolerance to withstand elevated temperatures without suffering a major reduction in selectivity and hence cannot be considered for the proposed aeration/vapor separation/condensation scheme.

The A/G Technology gas separation hollow fiber membranes are the only known membranes which can provide the requisite levels of both water vapor/air selectivity and water vapor effective permeability for the proposed concept. The selectivity of these membranes for water vapor to air is about 500 (temperature dependent) and the effective permeability for water vapor is estimated to be <u>between a minimum of 10 times, and possibly, 100 times higher</u> than the spiral wound, cellulosic membranes evaluated by Bend Research in their SBIR program entitled, "On-Board Water Generation for Military Vehicles" (5) conducted for the US Army Tank and Automotive Command.

The A/G Technology advanced permeable membranes have shown exceptional performance for both oxygen enrichment and nitrogen enrichment and thus, predictions of water vapor enrichment capability are believed to be reasonable. In the area of oxygen enrichment, the National Institutes of Health have funded a program (SBIR) to develop a <u>portable</u> unit for outpatient inhalation therapy. For nitrogen enrichment, an independent study commissioned by the US Air Force and conducted by Boeing Military Airplane Company (BMAC) has cited the advanced permeable membranes of A/G Technology to be the most advanced concept for On-Board Inert Gas generation Systems (OBIGGS) and have verified in comparative tests the 100-fold improvement in gas transport shown in Table 1 for A/G Technology membranes versus Dow membranes (2). Furthermore, in a recent communication BMAC has stated that the advanced permeable membrane

Table 1

BEST ESTIMATES OF COMPARATIVE GAS SEPARATION MEMBRANE INTRINSIC PROPERTIES

Company	Reference	Geometry	Temperature, (C)	Transport $[cm^3(STP)/cm^2$ sec cm-Hg] x 10^4		
				Oxygen	Nitrogen	Water Vapor
A/G Technology Corporation	(3)	HF	25	1 to 4	0.3 to 1	----
	Estimate	HF	25	----	----	600
	Estimate	**HF**	**70**	**5**	**2**	**1,500**
Bend, Cellulosic	(5)	SW	75	----	----	15
Enka AG, Cellulosic	(5)	FS	75	----	----	45
Olin, Cellulosic	(5)	FS	75	----	----	30
UOP Silicone	(2, 6)	SW	25	5 to 8.5	2.5 to 4.5	100 to 200
DOW, PMP	(7)	HF	25	0.04	0.01	----
Monsanto, Polysulfone	Estimate	HF	25	0.1 to 0.2	0.02 to 0.04	----

Notes:

1. HF = Hollow Fiber; SW = Spiral Wound; FS = Flat Sheet, amenable to spiral winding.
2. A/G Technology, Bend, UOP and Monsanto membranes are asymmetric. Others are dense.
3. Enka membrane sold under trade name of "Cuprophan".

cartridges "demonstrated to Boeing and Air Force representatives has satisfied any proof-of-concept and achievable performance improvement concerns" relating to cartridge scale-up for this demanding aircraft application (8).

IV. PROGRAM APPROACH FOR WFI PRODUCTION

A. Original Concept

The high water vapor transport characteristics through the A/G Technology gas separating membrane barrier can be exploited in combination with two exceptionally simple steps to render high purity water, potentially of WFI quality as stated in USP XXI (exclusive of final polishing steps, if required). The original WFI process scheme, shown in Figure 2, is able to process source waters with high organics and/or particulate loadings since an aeration step strips out organics and since hot gaseous vapors (primarily water vapor) are processed by the membrane cartridge rather than the source liquid eliminating the possibility of membrane fouling and/or fiber plugging. This WFI system approach consists of:

1. Aeration/Evaporation (utilizing waste heat) to Produce Water Vapor from any Source Water.
2. Gas Separation Membrane Processing to Produce a Water Vapor Enriched Stream, Free of Contamination.
3. Condensation/Deaeration to Produce WFI and Remove Non-Condensibles.

The aeration/evaporation step consists of sparging an air stream through the source water to saturate the air (working fluid) with water vapor. The water vapor content in the air is increased at elevated temperature and, hence, waste heat from utilities or power generators is utilized to raise the source water temperature to about 160 F. Highly volatile organics in the source water will be vaporized and partially removed by venting. Low vapor pressure organics will be concentrated in the air stripping column and removed via a drain valve. Unlike an evaporator, the heat exchanger surfaces are not exposed to evaporative conditions which enhance fouling and scaling from high concentrations of sparing soluble components. Feed water is added as needed to replenish the column, although it is of note that batchwise operation is also possible wherein aeration is performed as a conditioning step if the source water contains high levels of volatiles.

The water vapor saturated stream is fed into the hollow fiber membrane vapor enricher by a low pressure, centrifugal air blower at about 80 cm-Hg (16 psia). The hot vapor preferentially permeates the membrane and is enriched several hundred-fold on the down stream side of the cartridge. The water vapor enriched gas is passed through a condenser (e.g., air cooled) and collected in a condensate accumulator tank. Non-condensibles (essentially oxygen enriched air) are removed from the accumulator by a small vacuum pump and vented.

The gas membrane retentate (water vapor depleted air) is recycled through the system as the working fluid, with make-up air added as needed.

B. Improved WFI System Concept Based on Pervaporation

Given that the WFI system does not have to process highly contaminated source water or waste water as originally envisioned, the system can be simplified to run

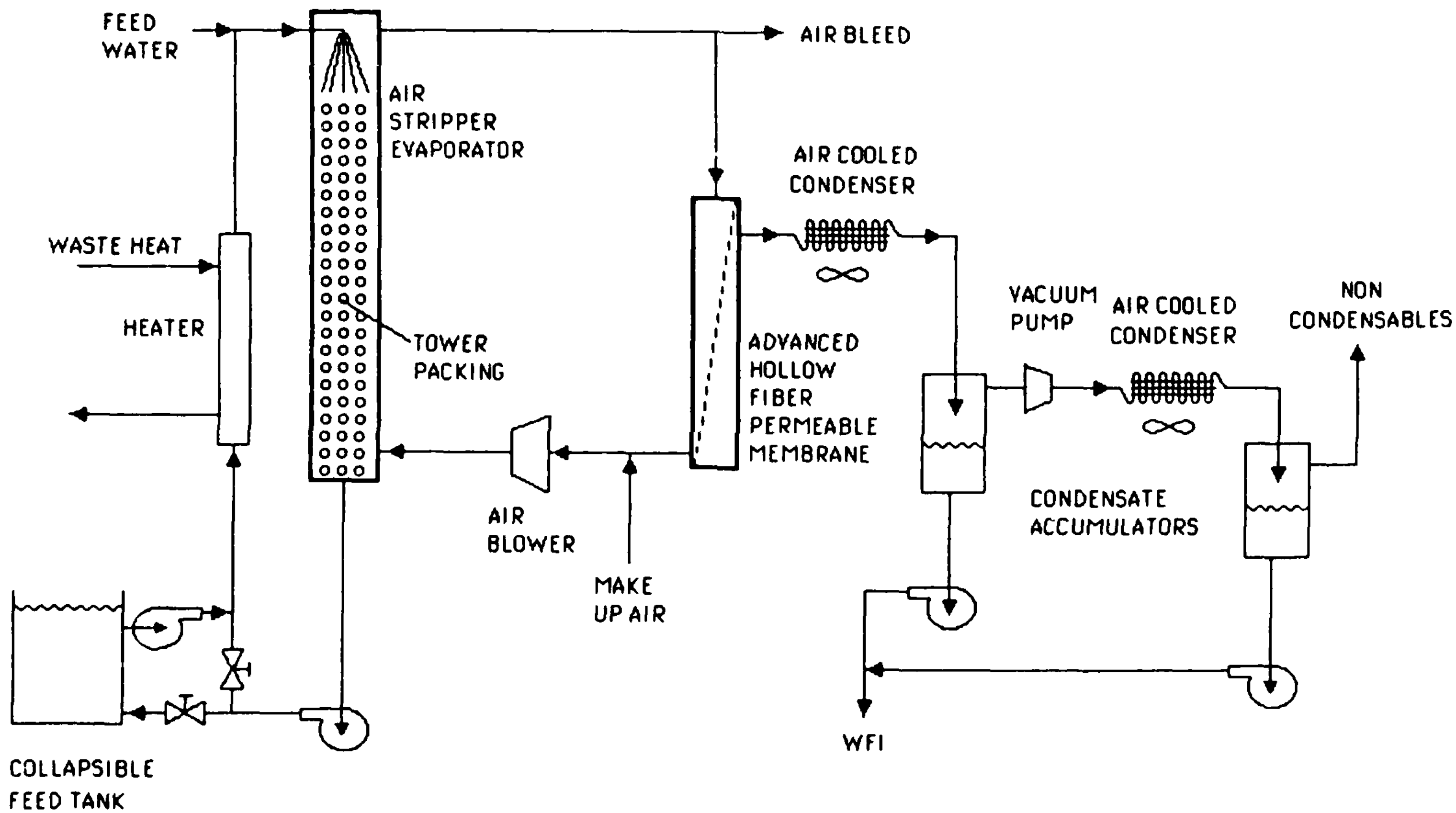

Figure 2. Flow Schematic of Original WFI Process

in a pervaporation mode. In this mode, hot water is fed on the lumen side of the hollow fiber membranes and water vapor is collected on the shell side of the membrane. A defect-free gas separation membrane is still required as the separating medium, as opposed to a reverse osmosis membrane which has separation layer imperfections, since the gas separation membrane will assure no bacteria or pyrogen passage into the WFI. Also, in the proposed pervaporation approach there is not the strong solute/membrane interaction found in reverse osmosis which leads to organics passage.

The improved WFI system concept is now further simplified to consist of:

1. Preheating (Utilizing Waste Heat) of the Source Water.
2. Pervaporation Across a Gas Separation Membrane to Produce a Water Vapor Enriched Stream, Free of Contamination.
3. Condensation/Deaeration to Produce WFI and Remove Non-Condensibles.

This approach eliminates the contact column and air blower required for aeration/evaporation and significantly reduces the size, weight and power of the vacuum pump utilized for non-condensible removal since minimum air is now transferred across the membrane. Moreover, as will be detailed below in the Results Section, the pervaporation approach provides a higher productivity of WFI per unit membrane area which further reduces system size and complexity. Depending on the available environmental control system, the WFI system may be easily integrated with available components.

A simplified schematic of the pervaporation system is shown in Figure 3. The source water is pumped through a heat exchanger countercurrently to waste heat and into the membrane cartridge. The hot water flows at low velocity and essentially atmospheric pressure through the hollow fiber membranes. Water vapor passes through the membrane and is drawn via a vacuum pump through an air cooled condenser and collected.

A review of Figure 2 versus Figure 3 shows the enhanced simplicity of the pervaporation approach.

C. Advantages of the Membrane Vapor Enricher Approaches to WFI Production

In addition to the proposed membrane vapor enricher approaches to WFI Production there are two other potential candidate systems:

1. Serial Membrane Filtration Consisting of:

   (a) Ultrafiltration Pretreatment
   (b) Reverse Osmosis Primary Treatment
   (c) Possibly Ion Exchange or Activated Carbon Polishing
   (d) Microfiltration for Final "Absolute" Bacteria and Particulate Removal

2. Evaporation and Condensation

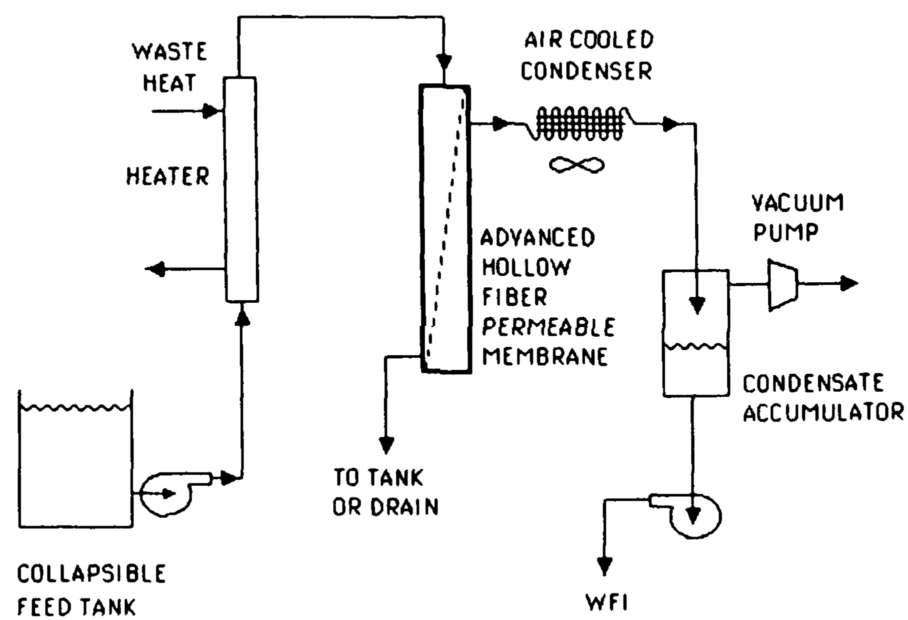

Figure 3. Flow Schematic of WFI Pervaporation Process

The advantages and disadvantages of these two approaches and of the proposed approaches to WFI production are detailed in Table 2.

Of major significance for combat situations is the non-fouling nature of the membrane vapor enricher concept. Logistic support for this system will be minimal. Furthermore, independent programs aimed at militarizing the advanced permeable membrane cartridge for the environmental extremes of MIL-STD-210 have been proposed as part of the qualification testing for Military Aircraft On-Board Inert Gas Generation Systems.

Table 2

COMPARATIVE APPROACHES TO WFI PRODUCTION

Technical Approach	Advantages	Disadvantages
Reverse Osmosis (RO) and Ultrafiltration (UF)	a) RO and UF are energy efficient. b) UF provides bulk removal of suspended solids and macromolecules. c) RO provides bulk removal of dissoved inorganics and some organics. d) Compact. e) RO provides good removal of bacteria and viruses.	a) UF considered as pre-treatment only. b) UF generally ineffective in dissolved solids separation. c) No absolute removal of bacteria and viruses. d) Some organics are not removed by RO and may even be negatively rejected (i.e., concentrated). e) RO performance is feed water dependent. f) RO cannnot operate at high temperatures to maintain system sterility. g) RO and/or UF membranes may be stored with chemical preservatives, requiring removal prior to use [Lostical Burden]. h) Multi-step filtration requires high level operators and more operator time. i) Requires ion exchange or activated carbon polishing for organics removal. j) Requires microfiltration for final "absolute" filtration of WFI.

Table 2 (Continued)

COMPARATIVE APPROACHES TO WFI PRODUCTION

Technical Approach	Advantages	Disadvantages
Evaporation	a) Can take advantage of waste heat. b) Good bulk removal of suspended sollids, dissolved solids, inorganics and some limited low boiling organics. c) Insensitive to high feed concentration levels.	a) Susceptible to product contamination by carry over of vapor or droplets. b) Requires waste heat to be energy efficient.
A/G Technology - Aeration/Vapor Separation/Condensation	a) High purity product (WFI) in simple process steps. b) Can utilize waste heat. c) Elimination of membrane fouling. d) Insensitive to feed water quality. e) No prefiltration requirement. f) Insensitive to feed concentration levels. Can process high salinity or contaminated water. g) Gas-vapor membrane offers excellent separation for non-strippable organics, bacteria, viruses and pyrogens. h) Most of the system is operated under low pressures. i) High throughput, compact, hollow fiber membrane cartridges.	a) Requires waste heat to be energy efficient. b) Relatively new process.

Table 2 (Continued)

COMPARATIVE APPROACHES TO WFI PRODUCTION

Technical Approach	Advantages	Disadvantages
A/G Technology - Aeration/Vapor Separaion/Condensation (Continued)	j) Technology transferrable to other water recovery/treatment problems. k) High temperature capability to maintain system sterility. l) Proposed gas separation membranes have indefinite dry storage. m) Proposed gas separation membranes have no break-in period and require no preconditioning.	
A/G Technology - Pervaporation/Condensation	a) High purity product (WFI) in simple process steps. b) *Can utilize waste heat.* c) When operated on "clean" feed water: -- No membrane fouling anticipated. -- No prefiltration requirement. d) Gas separation membrane offers excellent separation for organics, bacteria, viruses and pyrogens. e) System is operated under low pressures. f) High throughput, compact, hollow fibers. g) Technology transferrable to other water recovery/treatment problems. h) High temperature capability to maintain system sterility. i) Proposed gas separation membranes have indefinite dry storage. j) Proposed membranes have no break-in period and require no preconditioning.	a) Requires waste heat to be energy efficient. b) Relatively new process.

V. RESULTS AND DISCUSSION

A. General

In the Phase 1 program, the potential of this novel approach to WFI was assessed. The feasibility program consisted of the following six tasks:

Task 1: Test System Design and Assembly
Task 2: System Shakedown Testing with Tapwater
Task 3: Determine Membrane Water Vapor Transport Properties
Task 4: Assess WFI System Performance
Task 5: Extended Duration Test of WFI System
Task 6: Conceptual Design of Full-Scale REFLUPS

The original approach centered on contact of the membrane with an air/vapor mixture to alleviate the potential problems associated with processing "dirty" surface waters and waste waters through the hollow fiber membranes. When the feed water stream was defined to be of tap water quality, even in combat situations, the further simplified pervaporation approach became feasible and was also tested.

Comparison of the data achieved in testing both approaches shows the pervaporation technique to require less associated hardware, be simpler in operation and to have a higher throughput of WFI. Thus, the final program task, Conceptual Design of Full-Scale REFLUPS, considered only the optimum membrane vapor enricher approach.

B. Experimental

The test system was designed per the aeration/evaporation - membrane vapor enrichment- condensation scheme. With reference to Figure 2, the source water is heated to about 160 F and enters the air stripper/ evaporator near its top while the air enters the bottom of the stripper/ evaporator. The air stream is sparged through the source water to saturate the air (working fluid) with water vapor. The vapor exits the top of stripper/evaporator and, if present, highly volatile organics in the source water are vaporized and partially removed by the vent. Feed water is added as needed to replenish the column, although it is of note that batchwise operation is also possible wherein aeration is performed as a conditioning step if the source water contains high levels of volatiles.

The water vapor saturated stream then enters the hollow fiber membrane vapor enricher. The hot vapor preferentially permeates the membrane and is enriched several hundred-fold on the down stream side of the cartridge. The water vapor enriched gas is passed through a condenser (e.g., air cooled) and collected in a condensate accumulator tank. Non-condensibles from the first accumulator tank are fed by a vacuum pump into a second accumulator tank. The non-condensibles (essentially oxygen enriched air) are vented from the accumulator. The water condensed in the first accumulator tank can be recycled by a pump to the air stripper/evaporator (as a convenience in the test program) or removed as WFI.

The water condensed in the second accumulator tank is removed by a pump as WFI.

The gas membrane retentate (water vapor depleted air), is recycled through the system as the working fluid, with make-up air added as needed.

The test system includes appropriate flow, pressure and temperature instrumentation and is constructed of both high temperature plastics and stainless steel Furthermore, it is insulated to maintain process temperatures. This feasibility test system is not, however, designed to medical grade standards.

For operation in the pervaporation mode (Figure 3), the air blower was disconnected and the air stripper column was bypassed. Feed water was pumped directly from the feed tank through the heater and into the membrane cartridge. Since the system was not originally designed for this mode of operation and due to the increased productivity, the temperature of the water could not be maintained at the highest possible operating temperatures for long periods of time.

Throughout the test program, the air flowrate, water flowrate, water temperature, membrane inlet pressure, vacuum pump suction pressure and WFI accumulation rate were routinely monitored. Periodically, feed water and product water samples were collected and analyzed for pH, conductivity, suspended solids and for Total Organic Carbon (TOC) analysis. The TOC assays were performed by Enseco, Erco Laboratory, Cambridge, MA using Standard Method 415.2.

A simplified schematic of an A/G Technology AVIR™ hollow fiber membrane vapor enricher is shown in Figure 4. A multitude of hollow fibers are potted in parallel within a temperature resistant polysulfone housing. The housing outside diameter is 3 inches with the length variable depending on the required membrane surface area and the allowable pressure and/or temperature losses across the cartridge.

In this phase of the program, the cartridges were typically about 8 to 18 inches long with active surface areas of the hollow fiber membrane on the order of several square feet.

Feed, retentate and product ports are 1.5 inch sanitary-type (i. e., tri-clamp) connections.

The two synthetic feed streams originally believed to be representative of sources at potential field sites and able to provide an accurate reflection of the viability of the proposed process are:

1. High Salinity Water (Brackish, 10,000 ppm salts), and
2. MUST Hospital Waste Washwater Mixture (9).

The brackish water feed was prepared by dissolving the following salts in Needham, MA tap water:

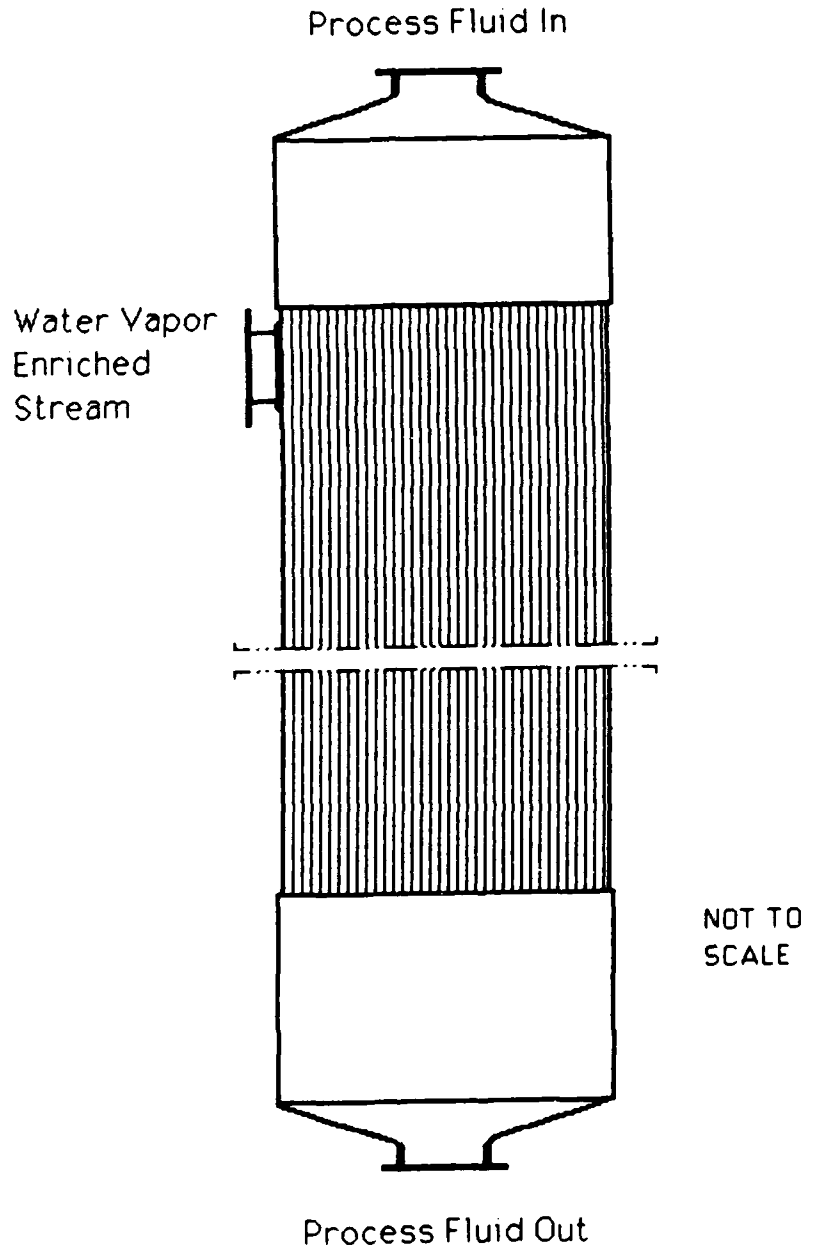

Figure 4. Simplified Sketch of A/G Technology Advanced Permeable Membrane Cartridge

Salt	Percentage
$CaSO_4$	11
$MgSO_4$	10
Na_2SO_4	55
$NaHCO_3$	18
NaCl	6
$Fe(NO_3)_3$	<1

These percentages are equivalent to a Colorado brackish water (9), however the overall salt concentration is about 5 times higher than typical for this surface water to provide a worst case test.

The components of the washwater in a Medical Unit, Self-Contained, Transportable (MUST) hospital are well documented (9) and synthetic formulations have been developed. In this program a synthetic feed incorporating the most readily available major components was prepared to test both process stability and product water quality under conditions of water reuse with a much more difficult source water than envisioned in actuality.

The MUST formulation prepared in tap water was:

Constituent	Concentration (mg/liter)
Detergent Type I (Tide)	650
Oil & Grease (Vegetable Oil)	200
Kaolinite Clay	150
Sour (Downey Fabric Softener)	116
Urea	20

Pervaporation testing was conducted with Needham, MA tap water.

C. Productivity

A series of tests was conducted to obtain WFI productivity data and to establish operating conditions for the Task 4 tests. Three parameters were varied during these transport data tests:

1. Operating Temperature;
2. Vapor Velocity through the Membrane; and,
3. Make up Air Volume added to the System.

Results of these tests are shown graphically in Figures 5, 6 and 7.

Operating temperature of the system was varied from 140 F to 160 F with a tap water feed. As expected the system capacity increases with increasing operating temperature and is quite sensitive to this parameter. This is because the permeability of the membrane increases with temperature and the pressure on the feed-side of the membrane; i.e., the driving force, is higher at higher temperatures. The improved WFI productivity demonstrated at a temperature of

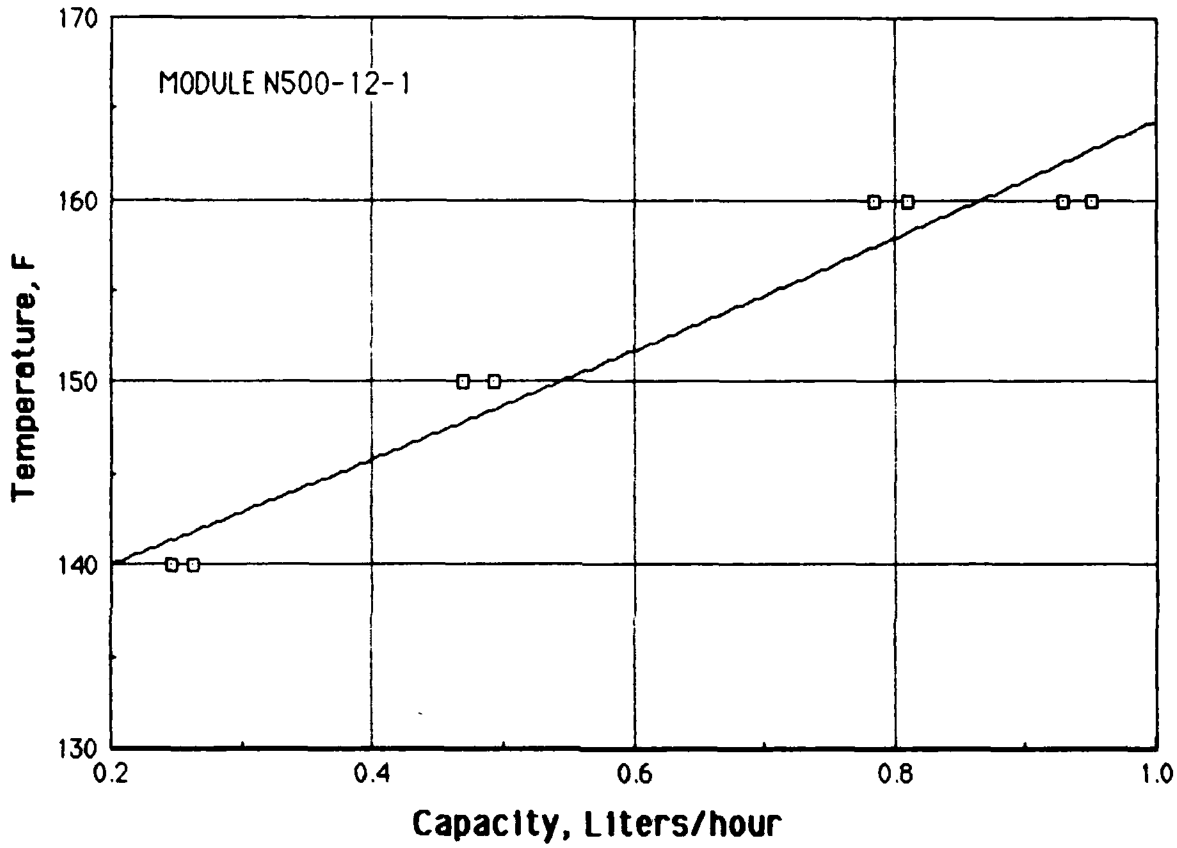

Figure 5. Phase 1 System WFI Production Capacity as a Function of Temperature

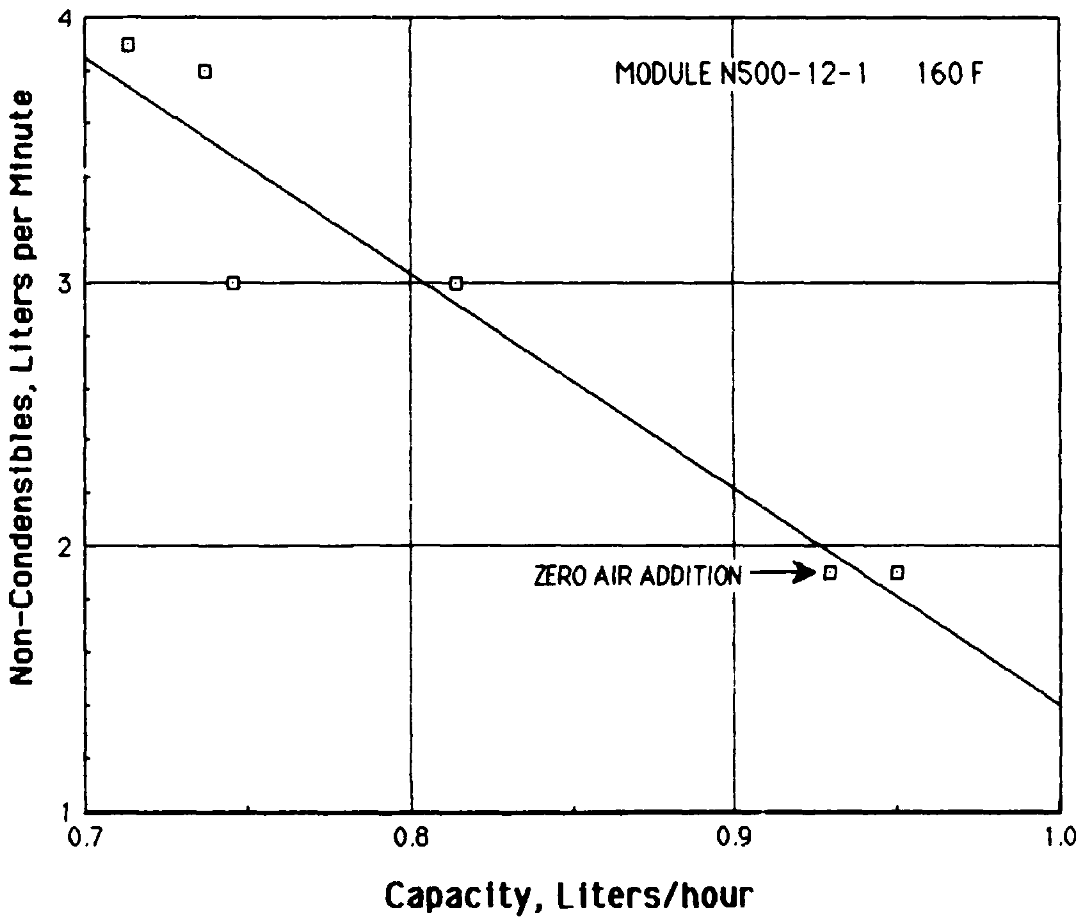

Figure 6. Phase 1 System WFI Production Capacity as a Function of the Non-Condensibles Flowrate

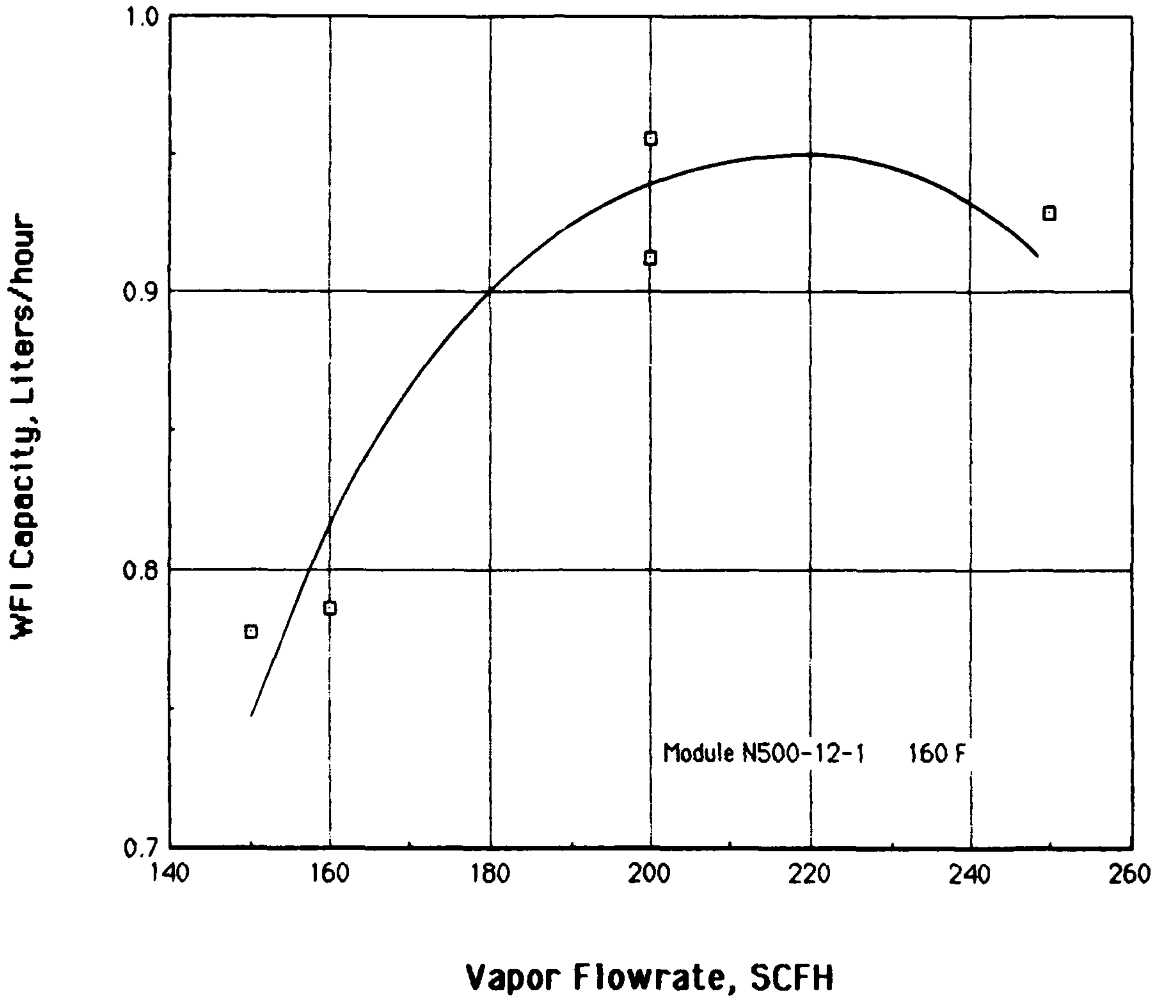

Figure 7. Phase 1 System Vapor Flowrate Versus WFI Production Capacity

160 F suggests that it is desirable to operate the WFI System at or above this temperature.

As shown in Figure 6, the system capacity varies inversely with the amount of air in the system. This curve is indicative of the impact of column pressure on the system capacity, however due to minor air leakage in the feasibility test unit, the trend of the data is considered more pertinent than the absolute values of the data.

The system capacity is relatively insensitive to air vapor velocity above a certain critical velocity, as indicated in Figure 7. At velocities above about 200 SCFH there was no significant change in WFI productivity.

Tests were also conducted to determine the effect of superheating the vapor entering the membranes. Although the superheating was effective in preventing a condensate film from forming on the membrane surface and increasing the water vapor permeability, it had the counter productive effect of increasing the air/water vapor ratio of the permeant.

An additional test was conducted with a short membrane cartridge to help understand the effect of cartridge geometry on productivity. The results of this test along with results with other cartridges are given below:

Cartridge Serial Number	Length (ft)	Area (ft^2)	Fiber Diameter (mm)	Productivity (liters/hr/ft^2)
5002-3A/1	1.5	8	0.9	0.13
N500-12/1	1.5	9.8	0.6	0.1
519-4B/2	0.84	3.8	0.8	0.19

Even though the oxygen permeabilities are similar for all of these membranes, the productivity varies. This is because operating conditions are altered as a function of cartridge geometry. In operation, most of the water vapor flows through the membrane in the first portion of the cartridge, resulting in the partial pressure of the water vapor being reduced rapidly as the flow proceeds along the fiber length. In general, it appears that shorter cartridge lengths and larger fiber diameters are beneficial.

D. WFI System Performance - Original Treatment Concept

1. General

The effectiveness of the proposed WFI production system is primarily a function of two factors:

1. Membrane Flux Stability (Non-Fouling), and
2. Product Water Quality.

To assess these factors, four tests were be conducted. Aeration conditions selected from prior extensive DOD-sponsored research (10), as well as, the preferred temperature determined from Task 3 were used as starting conditions.

Two tests were conducted with the synthetic brackish water and two tests with the MUST wastewater. Each test had a duration of 4 to 6 hour tests.

A summary of data from these tests and the tap water tests is given in Table 3. Data on column pressure and condenser pressure are included to indicate that the operating conditions were nearly the same for all three cases. The variation in liquid flow rate through the evaporative column is given to show the wide range of flow rates required to accommodate the foaming tendencies of the the test solutions.

The effectiveness of the membrane in removing all contaminants is dramatically illustrated in that the product water quality is nearly the same for all tests, independent of the composition of the feed.

2. Brackish Water Tests

A slight tendency towards foaming required a significant reduction in liquid flow rate through the evaporation column when processing the brackish water. Although the data suggest a slight reduction in capacity, compared to tap water tests, this is not significant considering the accuracy of the experimental measurements.

Two tests, the first of 6.5 hours duration and the second of 9.25 hours, were conducted During these tests capacity was stable and the equipment ran smoothly without any problems. Product water quality was measured during these tests as indicated in Table 3. The feed conductivity for the brackish water was 8,500 μmhos/cm with the WFI product water containing only 6 μmhos/cm. This represents a membrane rejection of over 99.9% for the salts which exceeds the salt retention of reverse osmosis membranes. In fact, as noted above, the system is not constructed to sanitary standards and the product quality indicated is not a true measure of the capability of the system, but rather data which can be used for comparative purposes, from one test to another. One can observe that the conductivity of the WFI product during a tap water processing run was also 6 mhos/cm, suggesting that some extractables were present in the non-sanitary piping.

3. MUST Tests

During the MUST tests the system operated well with stable performance. The quality of product water produced was the same or slightly better than produced during the brackish water tests . Flow through the evaporation column had to be reduced significantly (from 8,000 lb/hr-ft^2 in the tap water tests and 3,500 lb/hr-ft^2 in the brackish water tests) to 1,700 lb/hr-ft^2 for the MUST tests to prevent foaming. The lower flow resulted in a significant (30%) reduction in plant capacity.

Table 3

SUMMARY DATA FROM WFI SYSTEM

Variable	Tap Water	Brackish Water	MUST Water
Capacity (liters/hour)	1	0.93	0.67
Flux (liters/hour-ft^2)	0.09	0.08	0.06
Column Pressure (cm-Hg abs)	76	75	73
Condensor Pressure (cm-Hg abs)	13	13.6	9.5
Column Liquid Flow Rate (liters/min)	2.8	1.2	0.6
Water Quality:			
Feed Conductivity (micro mhos/cm)	50	8,500	3,700
Product Conductivity (micro mhos/cm)	6	6	3
Product Turbidity (NTU)	0.3	0.18	0.35
Product pH	6.5	6.3	6.5
Feed TOC (mg/liter)	NA	8.2	119
Product TOC (mg/liter)	NA	2.9	2.6

NA means Not Available.

In reducing the flow rates through the column to control foaming, a good flow distribution over the column packing could not be maintained. Therefore, more efficient packing materials were located and incorporated into the system at the end of the test program.

The conductivity of the MUST feed water was 3,700 μmhos/cm and again, a greater than 99.9% removal was achieved with the product water showing a conductivity of 3 μmhos/cm. Given that several tests had already occurred with a slightly higher conductivity measured in the product, it appears that the levels of extractables from the system components were lessening as a function of operating time.

It is significant to note that the TOC of the product water was only 2.6 mg/liter, essentially the same as the 2.9 mg/liter recorded with brackish water, eventhough the MUST feed water contained 119 mg/liter TOC. Using the brackish water test WFI as a baseline TOC for the system, the MUST product showed no TOC increase. Thus, it is envisioned that a sanitary-design system would produce acceptable quality WFI.

4. Extended Duration Test

To assess membrane performance as a function of time, a ten day (80 hour) test was conducted. Throughout the test system WFI productivity was stable and product quality showed minor improvement due to continual flushing of the system.

During the course of the extended duration test three major changes and a few minor were made to improve the test validity. First, communication from the Project Technical Monitor during the Project Review Meeting indicate that the WFI system would be operated on a feed of at least tap water quality, even in combat situations. Therefore, the last half of the extended duration test was conducted with a low salt content(1,000 ppm) brackish water. Next, the results of a preliminary conceptual design effort indicated that significant power savings could be obtained if the unit was operated with the evaporator column operating under vacuum. Thus, the last half of the tests were conducted in this mode. Finally, based on the results of the performance tests with MUST waste water, more efficient packing (Berl saddles) were obtained and installed in the evaporator column.

The changes in the evaporator column operation and packing must be considered as one reviews the extended duration test data. A tabular summary of the data is given in Table 4, with changes in feed stream and system operating parameters noted. It is important to note, that when a given set of operating conditions/system parameters were held constant the system provided stable WFI productivity. With the proper choice of parameters, the WFI productivity was doubled from the initial range of 0.7 to 0.8 liters/hour to about 1.5 liters/hour.

Table 4

EXTENDED DURATION TEST DATA

Parameter	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8	Day 9	Day 10 Start	Day 10 End
Run Time, Hours	7	8	8	9	8	8	8	9	8	6	2
Cumulative Time, Hours	7	15	23	32	40	48	56	65	73	79	81
Hot Water Temperature, F	160	160	160	160	160	160	160	160	160	160	160
Vapor In Temperature, F	149	149	145	144	148	147	152	152	154	152	152
Vapor Out Temperature, F	126	125	125	124	121	127	129	129	129	130	125
Column Pressure, cm Hg abs	72.6	72.6	39.6	39.6	38.1	39.1	39.4	39.4	38.4	39.1	75.2
Condensor Pressure, cm Hg abs	9.4	9.7	7.1	6.9	6.6	7.9	7.4	7.4	8.4	7.1	7.7
Blower Discharge Pressure, psig	1.1	0.7	-5.9	-5.9	-5.9	-5.9	-5.9	-5.9	-5.9	-5.9	0.7
Blower Flowrate, SCFH	284	277	159	154	106	143	158	158	157	158	276
Non-Condensible Flowrate, liters/minute	2.5	2.7	1.7	1.8	1.8	3	2.9	11	1.9	15	15
Productivity, liters/hour	0.68	0.66	0.78	0.79	0.77	0.78	1.49	1.49	1.46	1.51	0.76
Productivity, liters/hour/sq ft of membrane	0.06	0.06	0.07	0.07	0.07	0.07	0.13	0.13	0.13	0.13	0.07
Notes:	MUST Water through Day 5					1,000 PPM Brackish Water, Days 7 - 10					
						Day 6: New Column Saddles					
pH	6.5	6.5	6.5	6.5		6.5	6.3		6		5.5
Turbidity, NTU	0.4	0.38	0.4	0.34		0.25	0.28		0.18		0.13
Conductivity, mhos/cm	5	4	3	4.5		3	2		5.5		2.2
Membrane Serial #: 30162AB/1											
Membrane Area: 10,500 cm2											
Fiber ID: 0.55 mm											
Fiber Active Length: 10 cm											

E. WFI System Performance - Pervaporation Concept

A series of three tests was conducted to assess both the effectiveness of the simplified pervaporation approach and to determine the effect of temperature on WFI productivity. The data from these tests are shown graphically in Figure 8 along with comparative data from the original concept tests.

The pervaporation data clearly show a higher productivity than the vapor feed stream data at equivalent temperatures. For example, at a temperature of 149 F, the pervaporation approach shows over a 3-fold increase in productivity providing about 1.6 liters/hour WFI versus about 0.5 liters/hour WFI for the original approach.

It is also evident from Figure 8 that the slope of the productivity versus temperature curve for the pervaporation approach is much steeper than for the original approach, providing a projected 2.5 liters/hour WFI at 160 F for the small (11 ft^2 active area) test cartridge. The productivity in the pervaporation approach is also effected by the water recirculation rate through the cartridge. At higher recirculation rates, a higher average process temperature is maintained. The data presented in Figure 8 are at a recirculation rate of 2 liters/minute.

F. Conceptual Design of Full-Scale REFLUPS

The design of the full-scale system will be based on the less complex/higher productivity, pervaporation operating mode. The major elements of a WFI system based on this approach are:

1. Heat Exchanger
2. Water Pump
3. Membrane Cartridges
4. Condensor
5. Vacuum Pump
6. Condensate Pump
7. Associated Piping and Framework

Depending on the nature of the environmental control system, it may be possible to integrate the WFI system's heating and condensing steps with existing equipment. At this point in time it is premature to consider system automation and control in detail.

The system will be sized for 75 liters/hour (+/- 20%) WFI. It is believed that that this is a peak demand which will only be required on a periodic basis. Thus, the system may be operated at a low utilization rate, minimizing the importance of energy consumption.

Each element of the design is discussed below.

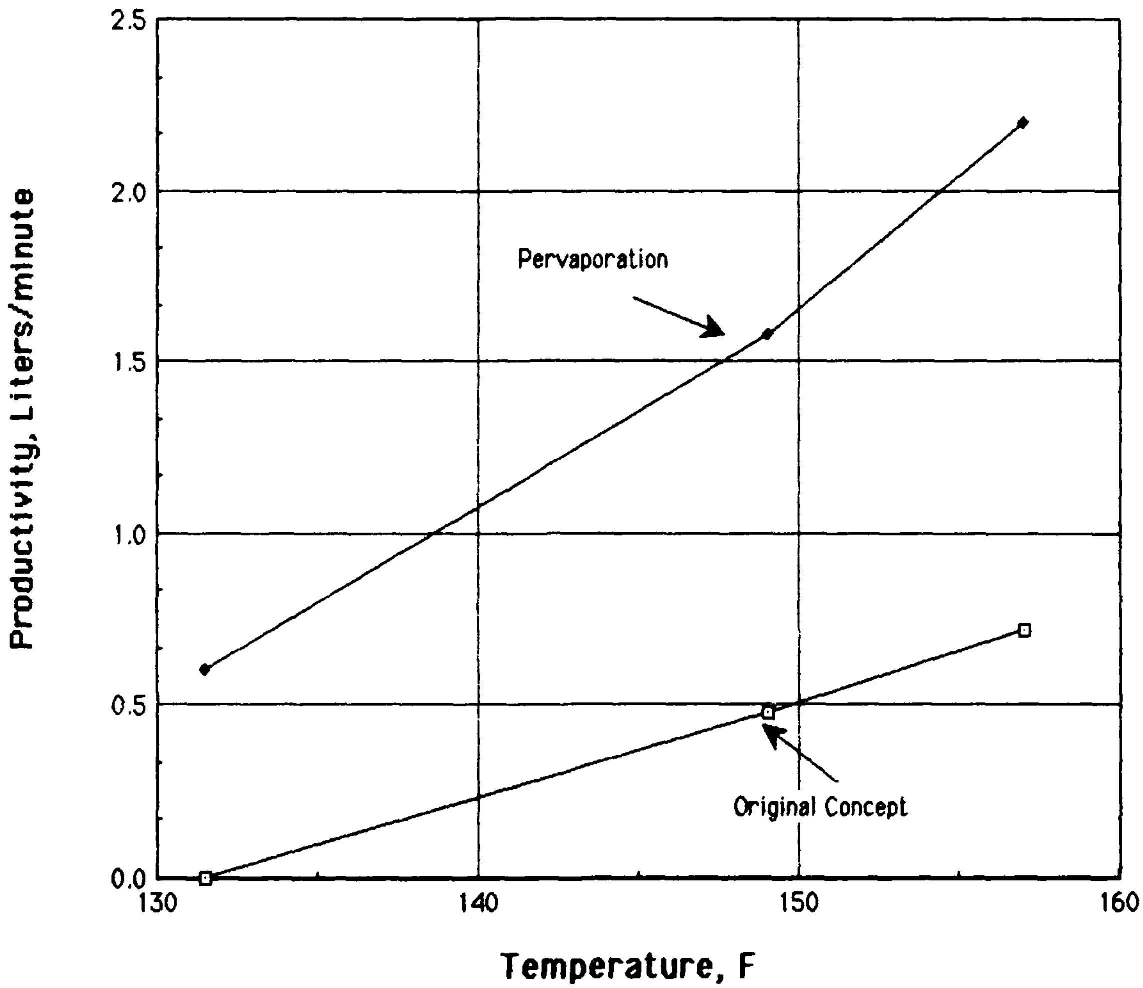

Figure 8. Comparative WFI Productivity as a Function of Temperature for the Two WFI Process Concepts

1. Heat Exchanger

It is assumed that waste heat in the form of hot water will be available at a temperature of 190 F. Approximately 17 gpm of 190 F water will be required to heat the 25 to 30 gpm of process water to the 160 F design temperature. The process water will be recycled within the system so that its inlet temperature into the heat exchanger will be about 145 F. Assuming a 20 F temperature loss across the heat exchanger for the waste heat (exit temperature 170 F) and a total transfer of 184,000 BTU/hr, the required heat exchanger surface area is on the order of 10 ft^2 (11).

Shell and tube stainless steel heat exchangers of this size are available from companies such as American Standard (12) with nominal dimensions of 4.5-in diameter x 40-in long. The weight of such a unit is 26 lbs.

2. Water Pump

The pump required to flow the process fluid through the heat exchanger will have a capacity of 30 gpm at a head of 10 psig. Numerous pumps are available in this capacity range. A typical pump has a 1/3 hp motor, is 6.5-in wide x 8.75-in high x 16.5-in long and weighs 34 lbs (13).

3. Membrane Cartridges

At an operating temperature of 160 F, the 11 ft^2 active area A/G Technology test cartridge is projected to produce 2.5 liters/hour WFI. Thus, to produce a nominal 75 liters/hour, a 30-fold increase in active membrane area is needed. The required area is therefore, 330 ft^2. Depending on the internal diameter of the hollow fiber membrane and the acceptable temperature and pressure drop across the membrane cartridge, from 2 to 4 cartridge elements will be required. Each element will have a diameter of 3-in and the length will range from about 25 to 50-in.

As a first approximation, it can be assumed that 3 cartridge elements, each 3-in diameter x 25-in long will be required. Each element will weigh about 4 lbs for a total membrane cartridge weight of about 12 lbs.

4. Condensor

The condenser may be either an air cooled or water cooled model. An air cooled unit has the advantage of not requiring cooling water hook-ups and availability but is somewhat larger and heavier than a water-cooled condenser. A suitably sized, finned air condenser (constructed to non-sanitary standards) has nominal dimensions of 30.5-in wide x 30.5-in high x 23-in long and weighs 184 lbs (14). This unit has a 0.75 hp fan and is rated at a working pressure of 300 psig (350 F). It is likely that a lighter weight unit, designed for the low operating pressures required in the WFI unit, can be identified in the future.

A water cooled condenser would be roughly equivalent to the heat exchanger sized for the heating of the incoming water. This shell and tube unit has dimensions of 4.5-in diameter x 40-in long and weights 26 lbs.

5. Vacuum Pump

The vacuum pump for the WFI system has a requirement of 0.02 SCFM (assuming 100% air saturated feed water) at 25-in Hg. This small flowrate can be achieved with a 1/7 hp unit such as a Thomas piston vacuum pump Model 607CA32 with dimensions of 5-in wide x 7-in high x 8-in long and a weight of 11 lbs (15).

6. Condensate Pump

The preferred method for filling bags with WFI has not been determined at this time. Either a condensate pump can be used or, if two collection vessels are provided with a vacuum break between them the bag filling can be by gravity feed. In the later case, a microporous filter would be required on the vacuum break line to prevent bacteria from entering the WFI collection container headspace.

For this conceptual design, it assumed that a pump will be incorporated into the system. The pump flowrate will be about 1 liter/minute and a positive displacement pump will be preferred. Miniature gear metering pumps are available in this capacity range with dimensions on the order of 2-in wide x 2-in high x 4.5-in long and weights of about 2 lbs (16). Pumps of this mature may be DC operated.

7. Associated Piping and Framework

The WFI system will have to be mounted on a frame with associated piping, hardware, insulation, instrumentation and controls. One or two collapsible tanks will be provided for WFI collection. It is beyond of the scope of this project to define these system elements. For an estimation of the total system weight, the weight of the associated piping ans framework is estimated to be 25% of the total weight of all other elements.

8. Summary of Physical Characteristics and Power Requirements

The physical characteristics and power requirements of the components detailed above are summarized in the following listing:

Component	Dimensions (in)	Weight (lbs)	Power (hp)
Heat Exchanger	4.5 diameter x 40 long	26	
Water Pump	6.5 wide x 8.75 high x 16.5 long	34	0.33
Membrane Cartridges	3 elements: 3 diameter x 25 long	12	
Condensor:			
(a) Air Cooled	30.5 wide x 30.5 high x 23 long	184	0.75
(b) Water Cooled	4.5 diameter x 40 long (condenser)	26	
	6.5 wide x 8.75 high x 16.5 long (pump)	34	0.33
Vacuum Pump	5 wide x 7 high x 8 long	11	0.14
Condensate Pump	2 wide x 2 high x 4.5 long	2	
Associated Equipment		36 - 67	

The estimated total dry weight of the unit, without instrumentation, is projected to be on the order of 180 to 336 lbs depending on the condenser type. The system volume is estimated to be between than 20 and 30 ft^3 with dimensions of about 2 ft wide x 2-ft x deep x 5-ft high with the water cooled condenser unit and about 2.5 wide x 2.5 deep x 5 ft high for the air cooled condenser unit. The unit, therefore, could easily be packaged into a 4-ft x 4-ft x 5-ft standard shipping container for transport by helicopter.

It is important to note that the above physical characteristics do not represent optimized equipment from the standpoints of weight and volume. It is very probable that more compact, lighter weight components are available.

The overall power requirement is estimated to be 0.8 to 1.25 hp for the air cooled and water cooled condenser options, respectively, assuming that waste heat is available. If waste heat is not available, the use of a heat pump is a viable option for a self-standing system. Assuming the heat pump has a coefficient of performance (COP) of 15, an additional power demand of about 5 hp is projected for the system.

VI. CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

As a result of this Phase 1 feasibility assessment program, the basic concept of using a newly developed, imperfection-free gas separation membrane for WFI production from a range of feed waters has been demonstrated. Specific conclusions are:

Process Scheme

1. Two process schemes were found to be acceptable in producing WFI. The original concept involves aeration and evaporation of the source water, vapor enrichment across the gas separation membrane and condensation of the vapor to form WFI. The second, even more simplified scheme, consists of heating the source water, processing the hot water through the gas separation membrane in a pervaporation mode and condensing the vapor to form WFI.

2. The pervaporation mode is preferred due to both its simplicity and its higher WFI productivity.

3. The key element of the WFI process, the hollow fiber permeable membrane gas separation cartridge, has been shown to process both liquid and vapor streams at high temperatures. The membrane's productivity when processing a "clean" (tap water equivalent) liquid stream is about 3 times its productivity on vapor streams due to the reduced non-condensible permeation across the membrane.

Process Performance

1. The vapor processing mode system performed equally well on Needham, MA tap water, synthetic brackish waters with both low (1,000 ppm) and high (10,000 ppm) salt contents and on a synthetic waste water consisting of chemicals found in MUST hospital effluents. By processing vapor, rather than the liquid, concentration polarization and membrane fouling due to constituents in the source water are eliminated.

2. When it became apparent that the source water would always be of tap water quality, the pervaporation mode became a viable alternative.

3. When different source waters are processed under similar operating parameters, the system productivity is similar.

4. Typical productivity at 160 F for the vapor enrichment operating mode is 0.08 liters/ft^2 of membrane/hour.

5. Typical productivity at 160 F for the pervaporation mode is 0.23 liters/ft^2 of membrane/hour.

6. Stable productivity was observed during an extended duration test.

7. Productivity has been shown to be partially a function of membrane cartridge hollow fiber internal diameter and active fiber length. In general larger diameters and shorter lengths produced higher productivity on a liters/unit membrane area/unit time basis.

7. Although the feasibility test system is not of a sanitary, medical-grade design, the product water was of excellent quality with a pH of 6.5, turbidity of 0.1 to 0.5 NTU, conductivity of 2 to 5 μmhos/cm and a TOC of 2 to 3 mg/liter.

8. When different source waters were processed, the product WFI was of consistent quality.

Physical Characteristics

1. The dry weight of a WFI system based on the pervaporation technique is projected to be on-the-order-of 180 to 340 lbs (dependent on condenser type, air or water cooled), without component optimization, instrumentation or automation.

2. The volume of a WFI system based on the pervaporation technique is projected to be on-the-order-of 20 to 30 ft^3, without component optimization, instrumentation or automation.

3. The power requirement for the pervaporation technique is projected to be between 0.8 to 1.25 hp with available waste heat. System designs for self-contained units incorporating a heat pump to eliminate the need for waste heat are projected to add minimal weight and volume and to increase the power requirement by about 5 hp.

B. Recommendations

Based on the results of the Phase 1 program, the following recommendations are offered:

1. The pervaporation mode should be optimized in terms of the physical dimensions: internal diameter and active length, of the hollow fiber membrane modules.

2. The pervaporation mode should be optimized in terms of the major operating parameters: water temperature and water velocity through the membrane cartridge.

3. A full-scale permeable membrane element should be tested in a breadboard (non-sanitary) system to verify WFI productivity and water quality on a larger scale.

4. The bread-board system should be operated on typical feed water (tap water) for an extended period of time to assess performance stability.

5. The conceptual design of a full-scale WFI system should be revised based on the additional experimental effort.

6. Consideration should be given to use of a heat pump to generate the heat required by the process in those cases where waste heat is not readily available.

7. Given the successful operation of the bread-board system, a manually operated, sanitary design, full-scale system should be designed, fabricated and tested.

8. The sanitary design system should then be instrumented, automated and tested prior to a final MIL-SPEC, MIL-STD design.

VII. PHASE 2 PROGRAM STATEMENT OF WORK

A. General

A seven task program is envisioned for the second phase of this program to provide optimization of WFI system operating parameters and permeable membrane physical characteristics and to develop long-term operating data. These tasks are:

Task 1: Modify Phase 1 Test System

Task 2: Optimize Membrane Cartridge Physical Characteristics

Task 3: Breadboard System Design and Assembly

Task 4: Optimize System Operating Parameters

Task 5: Extended Duration Test of Breadboard System

Task 6: Review of Self-Contained Heating Equipment Options

Task 7: Update Conceptual Design for Full-Scale REFLUPS

Each of these tasks is discussed below.

B. Task 1: Modify Phase 1 Test System

The test system designed and fabricated during Phase 1 will be modified to allow optimization of both the membrane cartridge physical characteristics and the pervaporation mode operating conditions. The system modifications will consist primarily of repiping to eliminate the aeration column and to reduce heat loss during processing.

C. Task 2: Optimize Membrane Cartridge Physical Characteristics

During the Phase 1 tests, the performance of the system in the vapor enrichment mode was observed to vary as a function of both hollow fiber membrane internal diameter and active length. In these tests, shorter cartridge lengths and larger fiber diameters proved beneficial to system productivity.

In order to test the effects of these parameters on the pervaporation mode, tests will be conducted with two different fiber internal diameters and two fiber lengths. The WFI productivity/unit membrane area will serve as a measure of the optimum cartridge design.

The fiber internal diameters will be within the range of 0.3 to 0.7 mm. The active fiber lengths to be tested will be within the range of 30 to 120 cm.

From within these ranges four tests will be conducted:

Test #	Fiber ID	Active Length
1	Small	Short
2	Small	Long
3	Large	Short
4	Large	Long

D. Task 3: Breadboard System Design and Assembly

Based on the results of Tasks 1 and 2, a breadboard system will be designed and assembled to test a full-scale membrane cartridge element. The test system WFI capacity will not be the 75 liters/hour required of a full-scale system since the heating load required to process this capacity will be excessive, resulting in heating equipment expenditures which are, perhaps, unjustified at this stage in the system development.

The breadboard system is projected to be from 1/5 to 1/3 of full-scale with a nominal output of 15 to 25 liters/hour WFI. This is an order-of-magnitude larger than the Phase 1 test system capacity. The system will be similar to the Figure 3 flow schematic with appropriate manual instrumentation and control. The heating source is envisioned to be an electric or gas heater, however, the use of a heat pump will also be considered.

As was the case with the Phase 1 test unit, this system will not be constructed to sanitary, medical grade standards. However, component selection will favor stainless steel and flourocarbon polymer materials of selection and other food/pharmaceutical grade materials such as silicone rubber.

E. Task 4: Optimize System Operating Parameters

Optimization of the system operating parameters will proceed with the preferred-design, full-scale membrane cartridge. The primary objective, assuming that waste heat will be available, is to maximize the productivity per unit membrane area.

A series of tests will be conducted to develop performance maps of WFI productivity as a function of temperature, water velocity through the cartridge and vacuum level drawn on the condenser.

It is envisioned that system performance will be assessed within the following ranges of these parameters:

Parameter	Range
Temperature	140 to 175 F
Water Flow/Product Flow Ratio	1 to 4
Vacuum Level	Determined by Temperature

Within this range of parameters, a minimum of 8 tests will be conducted to generate the performance map.

F. Task 5: Extended Duration Test of Breadboard System

Following selection of the optimum operating conditions, the breadboard unit will be operated for a minimum of 6 hours/day for 30 days to assess membrane flux stability and life. The source water throughout this test and the prior tasks will be Needham, MA tap water.

Samples will be collected daily for pH, and conductivity. On a weekly basis, feed and product samples will be collected for TOC and total solids assays.

G. Task 6: Review of Self-Contained Heating Equipment Options

As the WFI system is currently designed, a ready supply of waste heat must be available on-site. It may be desirable, however, to have a "self-standing" unit wherein the heat source is an integral component of the unit. To this end, a review of available vapor recompression/heat pump equipment will be conducted to ascertain the weight, volume and power requirements of a vapor recompression unit matched to the WFI system capacity.

H. Task 7: Update Conceptual Design for Full-Scale REFLUPS

The conceptual design of a full-scale REFLUPS developed in Phase 1 will be updated based on the results of Phase 2 testing. The basic system components will be reviewed and sized. The conceptual design will not be to medical class standards, however, pharmaceutical grade equipment will be sought. Assessment of on-line instrumentation to continuously monitor water quality is beyond the scope of this effort.

I. Program Reporting

Quarterly reports and a comprehensive Final Report will be submitted to the Army Project Officer.

J. Program Management

The program budget will be carefully monitored and the schedule reviewed regularly. The administrative tasks of coordinating staff assignments and equipment availability will be performed.

K. Phase 2 Program Schedule and Manpower Projection

The twelve month Phase 2 Program Schedule is detailed in Figure 9.

Figure 9. Projected Phase 2 Program Schedule (Months)

TASK DESCRIPTION	1	2	3	4	5	6	7	8	9	10	11	12
1. Modify Phase 1 Test System	====>											
2. Optimize Membrane Cartridge Physical Characteristics		====>										
3. Breadboard System Design and Assembly		================>										
4. Optimize System Operating Parameters						========>						
5. Extended Duration Test of Breadboard System								========>				
6. Review of Self-Contained Heating Equipment Options		======>										
7. Update Conceptual Design for Full-Scale REFLUPS										======>		
Travel to Belvoir R&D CEnter		**					**					
Reporting:												
Quarterly Reports				**			**			**		
Final Report											======>	
Program Management	==>											

The projected manpower requirement for Phase 2 is detailed in Table 5. For this phase, the total manpower requirement is 13.4 man-months. Details of the Army funding requirement are provided under separate cover.

L. Phase 3 Program

The third phase of development for the membrane water vapor enrichment system is to design and fabricate a manual-control full-scale unit to medical class standards. This unit may include on-line water quality instrumentation if the Army has already developed a suitable vendor list.

Extensive analytical work will be conducted to verify product water quality over long duration tests and system productivity will be assessed as a function of time. At the end of Phase 3, a detailed design for an automated WFI system will be developed

Table 5

PROJECTED PHASE 1 PROGRAM MANPOWER REQUIREMENTS (Man-Days)

Task Description	Principal Scientist	Program Manager	Project Engineer	Technician
1. Modify Phase 1 Test System	1.5	2	4	6.25
2. Optimize Membrane Cartridge Physical Characteristics	4	5	8	20
3. Breadboard System Design and Assembly	5	5	12.5	20
4. Optimize System Operating Parameters	7.5	8	12.5	20
5. Extended Duration Test of Breadboard System	4	5	7.5	15
6. Review of Self-Contained Heating Equipment Options	1	3	7.5	--
7. Update Conceptual design for Full-Scale REFLUPS	5	10	15	--
Travel to Belvoir R&D Center (2 Trips)	3	--	--	--
Reporting	7.5	15	10	--
Program Management	--	5	--	--
Totals	38.5	58	77	81.25

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